

WHAT IS CLAIMED IS:

1. A method for measuring at least one metal analyte in a halosilane source, comprising:

5 (a) collecting a first sample from a halosilane source, wherein the first sample comprises at least one halosilane having a formula $\text{Si}_u\text{Cl}_v\text{F}_w\text{Br}_x\text{I}_y\text{N}_z$, wherein u is 1 or 2; (v+w+x+y) is an integer between 1 and 4+2(u-1), inclusive; each of v, w, x, and y is an integer between 0 and 4+2(u-1), inclusive; z is an integer between 0 and 2u+1, inclusive; (v+w+x+y+z) is equal to 4+2(u-1); and each N is independently selected from the group consisting of hydrogen, methyl, methoxy, ethyl, ethoxy, propyl, propoxy, isopropyl, isopropoxy, n-butyl, butoxy, vinyl, and phenyl,

10 (b) contacting the first sample with an aqueous hydrofluoric acid solution, thereby producing a liquid reaction mixture,

15 wherein at least one chemical reaction occurs as a result of the contacting step, wherein the chemical reaction comprises the halosilane reacting with the aqueous hydrofluoric acid solution;

(c) evaporating liquid from the liquid reaction mixture, thereby producing a near-dry residue;

20 (d) mixing the near-dry residue with a take-up liquid, thereby producing a second sample; and

(e) analyzing the second sample for the presence of a detectable amount of at least one metal analyte.

2. The method of claim 1, wherein the first sample is a liquid.

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3. The method of claim 1, wherein the first sample is a gas.

4. The method of claim 1, wherein the first sample further comprises nitrogen gas or hydrogen gas.

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5. The method of claim 1, wherein the halosilane is a chlorosilane.

6. The method of claim 1, wherein each of w, x, and y is 0.
7. The method of claim 1, wherein the halosilane is selected from the group consisting of monochlorosilane, dichlorosilane, trichlorosilane, tetrachlorosilane, 5 methyltrichlorosilane, methyldichlorosilane, methylmonochlorosilane, dimethyldichlorosilane, dimethylchlorosilane, trimethylchlorosilane, hexachlorodisilane, triethoxyfluorosilane, phenyldiethoxychlorosilane, tetraethoxysilane, tetrafluorosilane, tetrabromosilane, and tetraiodosilane.
- 10 8. The method of claim 1, wherein the halosilane is selected from the group consisting of trichlorosilane, dichlorosilane, tetrachlorosilane, and tetrafluorosilane.
9. The method of claim 1, wherein the halosilane is trichlorosilane.
- 15 10. The method of claim 1, wherein the first sample comprises trichlorosilane and hydrogen.
11. The method of claim 1, wherein the first sample comprises trichlorosilane and nitrogen.
- 20 12. The method of claim 1, wherein the aqueous hydrofluoric acid solution comprises between about 49 wt% and 5 wt% hydrofluoric acid.
13. The method of claim 1, wherein the aqueous hydrofluoric acid solution 25 comprises between about 35 wt% hydrofluoric acid and 15 wt% hydrofluoric acid.
14. The method of claim 1, wherein the aqueous hydrofluoric acid solution comprises about 25 wt% hydrofluoric acid.
- 30 15. The method of claim 1, wherein the take-up liquid comprises nitric acid and hydrogen peroxide.

16. The method of claim 1, wherein the first sample is contacted with the aqueous hydrofluoric acid solution in the presence of a shield gas.
17. The method of claim 16, wherein the shield gas is nitrogen, argon, or helium.
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18. The method of claim 16, wherein the shield gas is nitrogen.
19. The method of claim 1, wherein the metal analyte is selected from the group consisting of iron, molybdenum, chromium, zinc, magnesium, tin, titanium, nickel, copper, aluminum, boron, phosphorous, calcium, sodium, manganese, vanadium, potassium, lithium, beryllium, gallium, germanium, arsenic, strontium, zirconium, niobium, cobalt, silver, cadmium, indium, antimony, barium, tantalum, thallium, lead, and bismuth.
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20. The method of claim 1, wherein the metal analyte is selected from the group consisting of boron, phosphorous, arsenic, antimony, germanium, iron, chromium, nickel, manganese, and molybdenum.
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21. The method of claim 1, wherein the metal analyte is selected from the group consisting of boron, phosphorus, and arsenic.
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22. The method of claim 1, wherein the analyzing step is performed using graphite furnace atomic absorption (GFAA) or inductively coupled plasma-mass spectrometry (ICP-MS).
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23. The method of claim 1, wherein the analyzing step is performed using dynamic reaction cell inductively coupled plasma-mass spectrometry (DRC ICP-MS).
24. The method of claim 1, wherein the analyzing step is performed using high resolution inductively coupled plasma-mass spectrometry.
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25. The method of claim 1, wherein the volume of the hydrofluoric acid solution is between about 400 and 500 ml, and the first sample is contacted with the aqueous hydrofluoric solution at a flow rate of between about 0.5 g/min and 1 g/min.

5 26. A method for measuring at least one metal analyte in a chlorosilane source, comprising:

(a) collecting a first sample from a chlorosilane source, wherein the first sample comprises at least one chlorosilane,

10 (b) contacting the first sample with an aqueous hydrofluoric acid solution, thereby producing a liquid reaction mixture,

wherein at least one chemical reaction occurs as a result of the contacting step, wherein the chemical reaction comprises the chlorosilane reacting with the aqueous hydrofluoric acid solution;

15 (c) evaporating liquid from the liquid reaction mixture, thereby producing a near-dry residue;

(d) mixing the near-dry residue with a take-up liquid, thereby producing a second sample; and

(e) analyzing the second sample for the presence of a detectable amount of at least one metal analyte.

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27. The method of claim 26, wherein the chlorosilane has a formula $\text{Si}_u\text{Cl}_v\text{N}_z$, wherein u is 1 or 2; v is an integer between 1 and $4+2(u-1)$, inclusive; z is an integer between 0 and $2u+1$, inclusive; $(v+z)$ is equal to $4+2(u-1)$; and each N is independently selected from the group consisting of hydrogen, iodo, bromo, fluoro, methyl, methoxy, ethyl, ethoxy, propyl, propoxy, isopropyl, isopropoxy, n-butyl, butoxy, vinyl, and phenyl.

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28. The method of claim 27, wherein each N is independently selected from the group consisting of hydrogen, methyl, methoxy, ethyl, ethoxy, propyl, propoxy, isopropyl, isopropoxy, n-butyl, butoxy, vinyl, and phenyl.

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29. The method of claim 26, wherein the chlorosilane is selected from the group consisting of trichlorosilane, dichlorosilane, and tetrachlorosilane.

30. The method of claim 26, wherein the aqueous hydrofluoric acid solution
5 comprises about 25 wt% hydrofluoric acid.

31. The method of claim 26, wherein the take-up liquid comprises nitric acid and hydrogen peroxide.

10 32. The method of claim 26, wherein the first sample is contacted with the aqueous hydrofluoric acid solution in the presence of a shield gas.

33. The method of claim 26, wherein metal analyte is selected from the group consisting of boron, phosphorous, arsenic, antimony, germanium, iron, chromium,
15 nickel, manganese, and molybdenum.

34. The method of claim 26, wherein the analyzing step is performed using graphite furnace atomic absorption (GFAA) or inductively coupled plasma-mass spectrometry (ICP-MS).

20 35. A system for measuring at least one metal analyte from a halosilane source, comprising:
a halosilane source comprising at least one halosilane having a formula $\text{Si}_u\text{Cl}_v\text{F}_w\text{Br}_x\text{I}_y\text{N}_z$, wherein u is 1 or 2; (v+w+x+y) is an integer between 1 and 4+2(u-1),
25 inclusive; each of v, w, x, and y is an integer between 0 and 4+2(u-1), inclusive; z is an integer between 0 and 2u+1, inclusive; (v+w+x+y+z) is equal to 4+2(u-1); and each N is independently selected from the group consisting of hydrogen, methyl, methoxy, ethyl, ethoxy, propyl, propoxy, isopropyl, isopropoxy, n-butyl, butoxy, vinyl, and phenyl,

30 at least one reaction system coupled to said halosilane source, wherein a first sample comprising the halosilane is collected from the halosilane source, wherein the

first sample and with an aqueous hydrofluoric acid solution are contacted in the reaction system to produce a liquid reaction mixture,

an evaporator, wherein the evaporator is used to evaporate liquid from the liquid reaction mixture to produce a near-dry residue, wherein the near-dry residue is mixed with a take-up liquid to produce a second sample, and

5 a metal analyte detector, wherein the metal analyte detector is used to analyze the second sample for the presence of a detectable amount of at least one metal analyte.

10 36. The system of claim 35, wherein the halosilane source is selected from the group consisting of a halosilane canister, a halosilane bulk storage tank, halosilane supply line, and a deposition chamber.

15 37. The system of claim 35, wherein the halosilane source is a chlorosilane source comprising at least one chlorosilane.

38. The system of claim 35, wherein the reaction system comprises:

a sample introduction line to carry the first sample,

a shield gas supply line,

20 a shield gas functional line,

wherein the shield gas supply line and the shield gas functional line are to carry a shield gas,

wherein the shield gas functional line has a longitudinal axis that is parallel to the longitudinal axis of the sample introduction line, and the sample shield gas functional line jackets at least a portion of the sample introduction line,

wherein the jacketed portion of the sample introduction line comprises an open end and a continuing end, and

25 30 wherein the shield gas functional line comprises a first end that is sealed to the continuing end of the portion of the sample introduction line and an open end,

a connector that connects the shield gas supply line and the shield gas functional line,

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wherein the open end of the jacketed portion of the sample introduction line and the open end of the shield gas functional end are on the same side relative to the connector, and

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an impinger comprising a reaction vessel and the aqueous hydrofluoric acid solution therein,

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wherein the open end of the shield gas functional line and the open end of the jacketed portion of the sample introduction line are positioned below the surface of the aqueous hydrofluoric acid solution, and

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wherein the open end of the shield gas functional line and the open end of the sample introduction line are positioned relative to one another such that when the shield gas and the first sample are carried through the reaction system, the shield gas is capable of shielding the first sample when the first sample is contacted with the aqueous hydrofluoric acid solution, thereby producing the liquid reaction mixture.

39. The system of claim 38, wherein at least one of the sample introduction line, the shield gas supply line, the shield gas functional line, the connector, or the reaction vessel comprises polytetrafluoroethylene (PTFE).

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40. The system of claim 38, wherein at least one of the sample introduction line, the shield gas supply line, the shield gas functional line, the connector, or the reaction vessel comprises perfluoroalkoxy (PFA).

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41. The system of claim 38, wherein the connector is a tee connector.

42. The system of claim 38, wherein the aqueous hydrofluoric acid solution comprises between about 49 wt% and 5 wt% hydrofluoric acid.

43. The system of claim 38, wherein contacting the first sample with the aqueous hydrofluoric acid solution in the impinger further produces an exhaust gas, and wherein the reaction system further comprises an abatement line and an abatement unit, wherein the abatement line is capable of carrying the exhaust gas from the impinger to the abatement unit, and the abatement unit comprises an abatement vessel and an aqueous caustic solution.

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44. The system of claim 35, wherein the detector uses graphite furnace atomic absorption (GFAA) or inductively coupled plasma-mass spectrometry (ICP-MS).

45. The system of claim 35, wherein the detector is capable of detecting at least one metal analyte selected from the group consisting of boron, phosphorous, arsenic, antimony, germanium, iron, chromium, nickel, manganese, and molybdenum.

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46. The system of claim 35, wherein the detector is capable of detecting less than about one ppbw of at least one metal analyte.

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47. The system of claim 35, wherein each of w, x, and y is 0.

48. A system for measuring at least one metal analyte from a chlorosilane source, comprising:

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a chlorosilane source comprising at least one chlorosilane, at least one reaction system coupled to said chlorosilane source, wherein a first sample comprising the chlorosilane is collected from the chlorosilane source, wherein the first sample and with an aqueous hydrofluoric acid solution are contacted in the reaction system to produce a liquid reaction mixture,

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an evaporator, wherein the evaporator is used to evaporate liquid from the liquid reaction mixture to produce a near-dry residue, wherein the near-dry residue is mixed with a take-up liquid to produce a second sample, and

a metal analyte detector, wherein the metal analyte detector is used to analyze the second sample for the presence of a detectable amount of at least one metal analyte.

5 49. The system of claim 48, wherein the chlorosilane has a formula $\text{Si}_u\text{Cl}_v\text{N}_z$, wherein u is 1 or 2; v is an integer between 1 and $4+2(u-1)$, inclusive; z is an integer between 0 and $2u+1$, inclusive; $(v+z)$ is equal to $4+2(u-1)$; and each N is independently selected from the group consisting of hydrogen, iodo, bromo, fluoro, methyl, methoxy, ethyl, ethoxy, propyl, propoxy, isopropyl, isopropoxy, n-butyl, butoxy, vinyl, and phenyl.

10 50. The system of claim 49, wherein each N is independently selected from the group consisting of hydrogen, methyl, methoxy, ethyl, ethoxy, propyl, propoxy, isopropyl, isopropoxy, n-butyl, butoxy, vinyl, and phenyl.

15 51. A system for measuring at least one metal analyte in a halosilane source, comprising:

20 (a) a means for collecting a first sample from a halosilane source, wherein the first sample comprises at least one halosilane having a formula $\text{Si}_u\text{Cl}_v\text{F}_w\text{Br}_x\text{I}_y\text{N}_z$, wherein u is 1 or 2; $(v+w+x+y)$ is between 1 and $4+2(u-1)$, inclusive; each of v, w, x, and y is between 0 and $4+2(u-1)$, inclusive; z is between 0 and $2u+1$; $(v+w+x+y+z)$ is equal to $4+2(u-1)$; and each N is independently selected from the group consisting of hydrogen, methyl, methoxy, ethyl, ethoxy, propyl, propoxy, isopropyl, isopropoxy, n-butyl, butoxy, vinyl, and phenyl,

25 (b) a means for contacting the first sample with an aqueous hydrofluoric acid solution, thereby producing a liquid reaction mixture,

wherein at least one chemical reaction occurs as a result of the contacting step, wherein the chemical reaction comprises the chlorosilane reacting with the aqueous hydrofluoric acid solution;

30 (c) a means for evaporating liquid from the liquid reaction mixture, thereby producing a near-dry residue;

(d) a means for mixing the near-dry residue with a take-up liquid, thereby producing a second sample; and

(e) a means for analyzing the second sample for the presence of a detectable amount of at least one metal analyte.

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52. The system of claim 51, wherein each of w, x, and y is 0.

53. A system for sampling for at least one metal analyte in a halosilane supply, comprising:

10 (a) a halosilane supply line to transport at least one halosilane having a formula $\text{Si}_u\text{Cl}_v\text{F}_w\text{Br}_x\text{I}_y\text{N}_z$, wherein u is 1 or 2; (v+w+x+y) is between 1 and 4+2(u-1), inclusive; each of v, w, x, and y is between 0 and 4+2(u-1), inclusive; z is between 0 and 2u+1; (v+w+x+y+z) is equal to 4+2(u-1); and each N is independently selected from the group consisting of hydrogen, methyl, methoxy, ethyl, ethoxy, propyl, propoxy, isopropyl, isopropoxy, n-butyl, butoxy, vinyl, and phenyl,

15 (b) a sampling unit coupled with said halosilane supply line, said sampling unit being capable of:

(i) collecting a first sample from the halosilane supply line, wherein the first sample comprises the halosilane; and

20 (ii) contacting the first sample with an aqueous hydrofluoric acid solution, thereby producing a liquid reaction mixture,

wherein at least one chemical reaction occurs as a result of the contacting step, wherein the chemical reaction comprises the halosilane reacting with the aqueous hydrofluoric acid.

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54. The system of claim 53, wherein each w, x, and y is 0.

55. The system of claim 53, wherein said sampling unit, comprises:

a sample introduction line to carry the first sample;

30 a shield gas supply line;

a shield gas functional line,

wherein the shield gas supply line and the shield gas functional line are to carry a shield gas,

5 wherein the shield gas functional line has a longitudinal axis that is parallel to the longitudinal axis of the sample introduction line, and the sample shield gas functional line jackets at least a portion of the sample introduction line,

10 wherein the jacketed portion of the sample introduction line comprises an open end and a continuing end, and wherein the shield gas functional line comprises a first end that is sealed to the continuing end of the portion of the sample introduction line and an open end;

15 a connector that connects the shield gas supply line and the shield gas functional line,

wherein the open end of the jacketed portion of the sample introduction line and the open end of the shield gas functional end are on the same side relative to the connector;

20 an impinger comprising a reaction vessel and the aqueous hydrofluoric acid solution therein,

wherein the open end of the shield gas functional line and the open end of the jacketed portion of the sample introduction line are positioned below the surface of the aqueous hydrofluoric acid solution, and

25 wherein the open end of the shield gas functional line and the open end of the sample introduction line are positioned relative to one another such that when the shield gas and the first sample are carried through the reaction system, the shield gas is capable of shielding the first sample when the first sample is contacted with the aqueous hydrofluoric acid solution, thereby producing the liquid reaction mixture and at least one exhaust gas;

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an abatement line; and
an abatement unit comprising an abatement vessel and an aqueous caustic solution,
wherein the abatement line is capable of carrying the exhaust
5 gas from the impinger to the abatement unit.

56. A system, comprising:

a processing tool to process workpieces,
a halosilane supply line coupled to said processing tool, said halosilane supply
10 line to supply at least one of trichlorosilane, dichlorosilane, tetrachlorosilane, and
tetrafluorosilane for said processing of semiconductor wafers, wherein the
chlorosilane,

15 a sampling unit coupled to the tool and halosilane supply line, said sampling
unit being capable of:

(i) collecting a first sample from the halosilane supply line, wherein
the first sample comprises at least one of trichlorosilane,
dichlorosilane, tetrachlorosilane, and tetrafluorosilane; and

(ii) contacting the first sample with an aqueous hydrofluoric acid
solution, thereby producing a liquid reaction mixture,

20 wherein at least one chemical reaction occurs as a result of the
contacting step, wherein the chemical reaction
comprises the halosilane reacting with the aqueous
hydrofluoric acid.

25 57. The system of claim 56, wherein the first sample comprises trichlorosilane.

58. The system of claim 56, further comprising a process controller coupled to the
processing tool, the halosilane supply line, and the sampling unit, said process
controller unit to control an operation of at least one of the processing tool, the
30 halosilane supply line, and the sampling unit.

59. The system of claim 56, wherein the sampling unit further, comprises:

a sample introduction line to carry the first sample;
a shield gas supply line;
a shield gas functional line,
wherein the shield gas supply line and the shield gas functional
5 line are to carry a shield gas,
wherein the shield gas functional line has a longitudinal axis
that is parallel to the longitudinal axis of the sample
introduction line, and the sample shield gas functional
line jackets at least a portion of the sample introduction
10 line,
wherein the jacketed portion of the sample introduction line
comprises an open end and a continuing end, and
wherein the shield gas functional line comprises a first end that
is sealed to the continuing end of the portion of the
sample introduction line and an open end;
15 a connector that connects the shield gas supply line and the shield gas
functional line,
wherein the open end of the jacketed portion of the sample
introduction line and the open end of the shield gas
20 functional end are on the same side relative to the
connector; and
an impinger comprising a reaction vessel and the aqueous hydrofluoric
acid solution therein,
wherein the open end of the shield gas functional line and the
open end of the jacketed portion of the sample
introduction line are positioned below the surface of the
aqueous hydrofluoric acid solution, and
25 wherein the open end of the shield gas functional line and the
open end of the sample introduction line are positioned
relative to one another such that when the shield gas and
the first sample are carried through the reaction system,
the shield gas is capable of shielding the first sample

when the first sample is contacted with the aqueous hydrofluoric acid solution, thereby producing the liquid reaction mixture.

5 60. The system of claim 56, wherein the workpieces are semiconductor wafers.